

Peroxide cured silicone rubber

Optimization of property

combinations with Aktisil Q

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1 Introduction

High-temperature curing (HTV) solid silicone rubbers are used in molded articles, e.g. in the automotive industry, in technical molding articles etc., as well as in extruded articles, e.g. cable applications and hoses.

The versatile application possibilities of these polymers are based on:

- easy processing
- excellent general mechanical properties
- outstanding compression set
- very good thermal and chemical resistance

Hoffmann Mineral offers Aktisil Q, a functionalized Neuburg Siliceous Earth that has been specially developed for use in silicone rubber.

Aktisil Q facilitates the processing of silicone rubber, since on the one hand it reduces or eliminates tack - depending on the dosage - and on the other hand it increases the collapse resistance of profiles during extrusion.

Apart from the markedly improved oil resistance, Aktisil Q scores especially with an outstanding compression set.

Normally, an improvement in compression set means a simultaneous deterioration of, for example, tear resistance, since these are two opposing properties.

Aktisil Q, on the opposite side, at low dosages leads to a constantly high, in some cases even to an additionally improved tear resistance, as the following results show.

2 Experimental

2.1 Formulation components and mixing

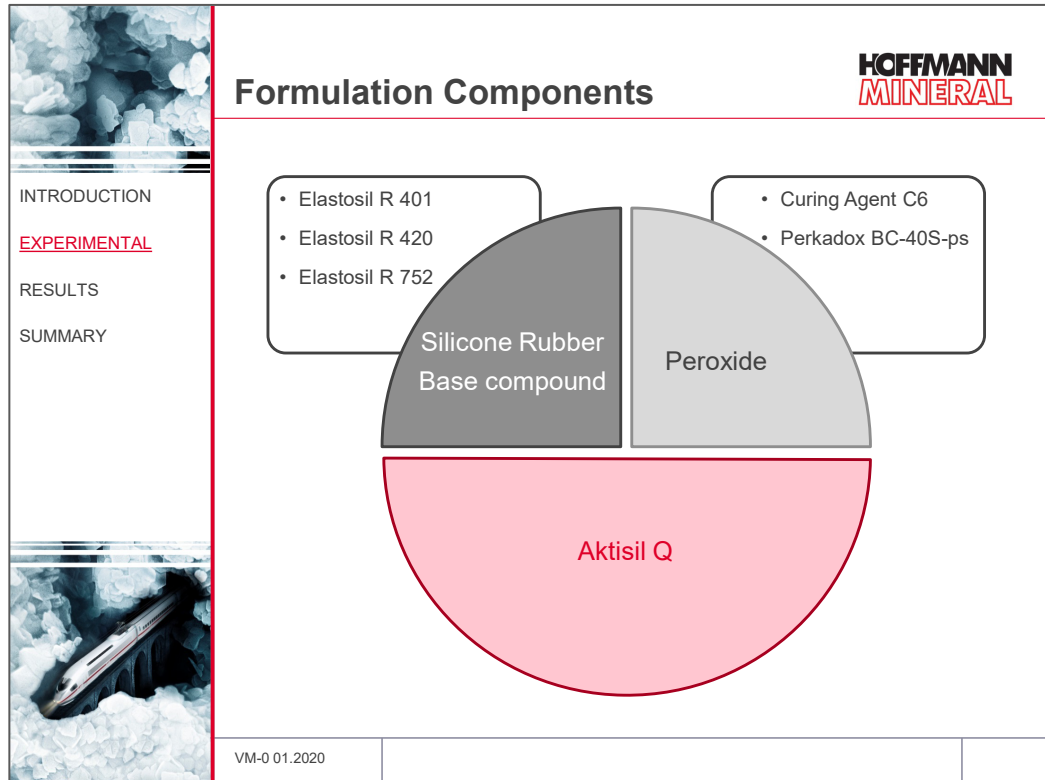


Fig.1

For this test series Aktisil Q was tested in three different polymers with two different peroxides each (Fig. 1).

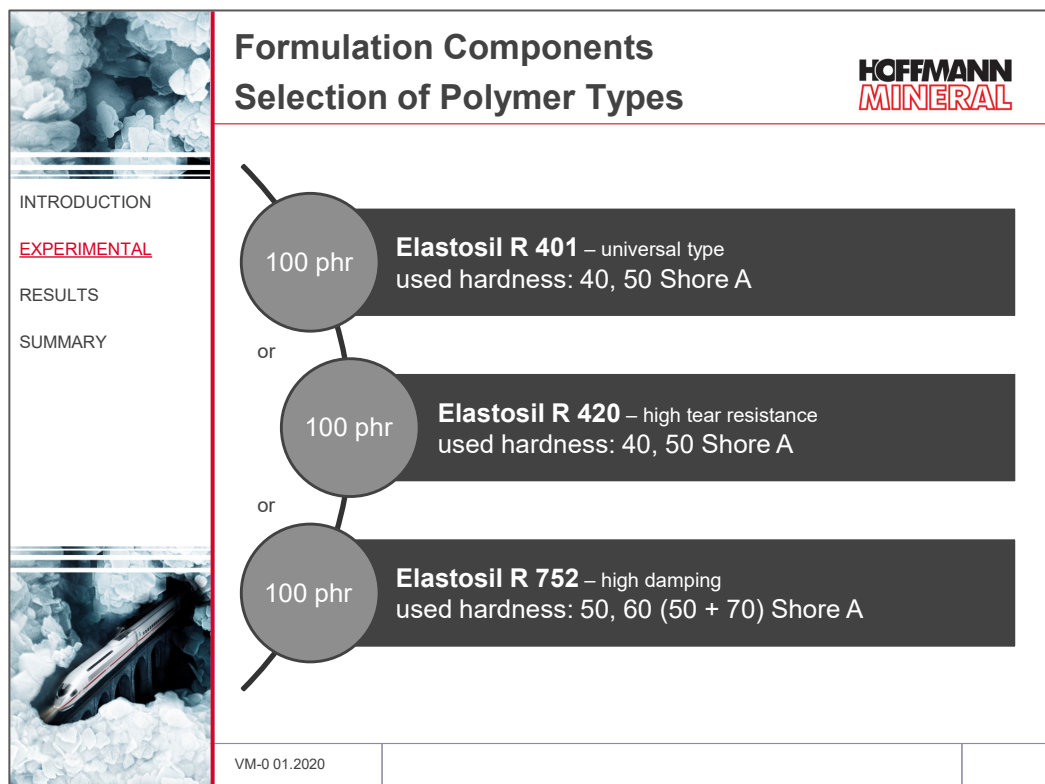


Fig. 2

In addition to the universal type Elastosil R 401, which has already been used in numerous tests at Hoffmann Mineral, Elastosil R 420, a type for high tear resistance, and Elastosil R 752, a damping polymer type, were used (Fig. 2).

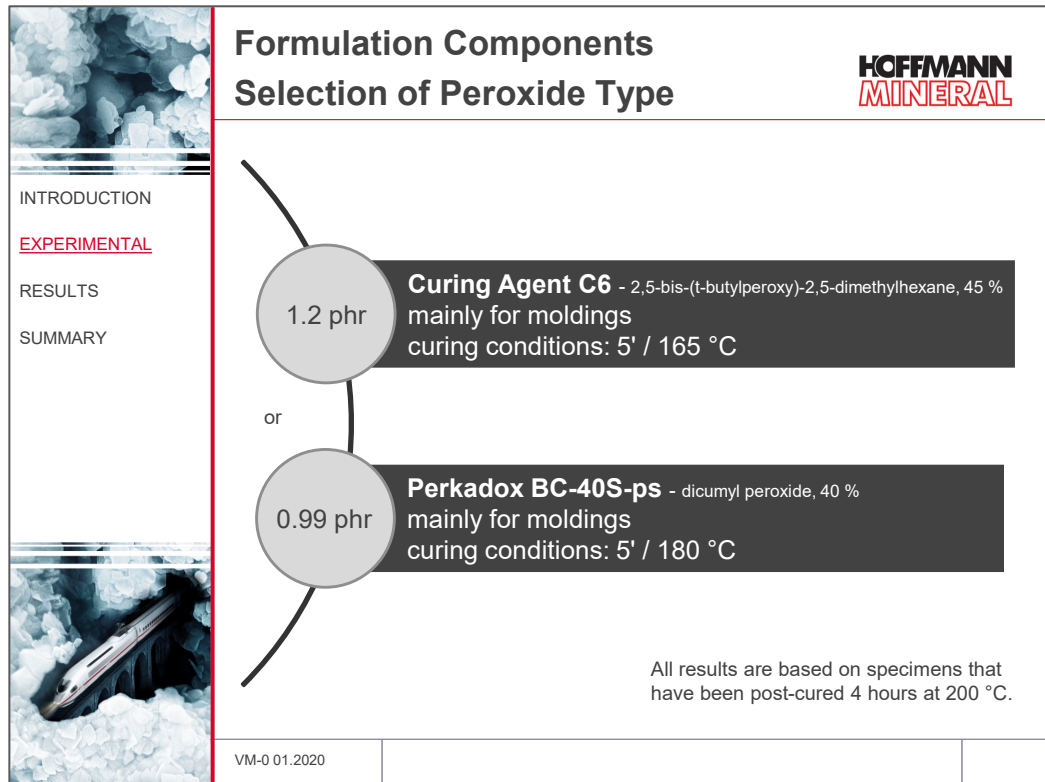


Fig. 3

With Curing Agent C6 (2,5-bis-(t-butylperoxy)-2,5-dimethylhexane) and Perkadox BC-40S-ps (dicumyl peroxide) two peroxides commonly used for molded articles were used. Fig. 3 shows the dosages and curing conditions used for each peroxide.

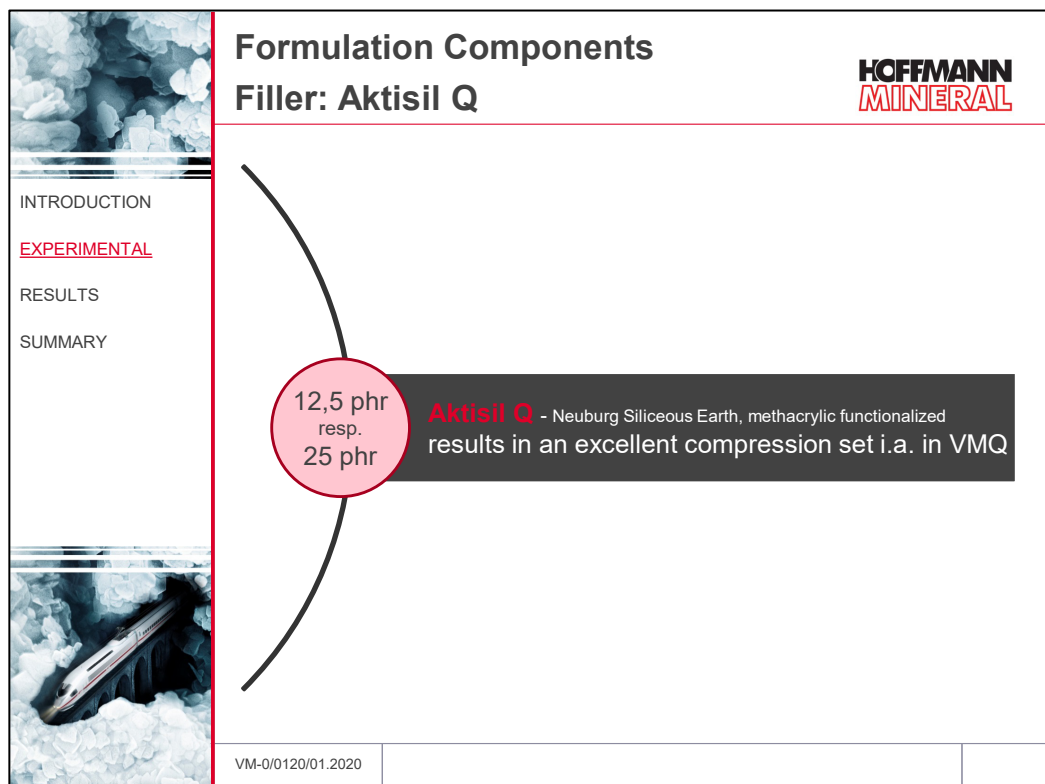


Fig. 4

The methacrylic funktional treated Neuburg Siliceous Earth Aktisil Q was applied in two different loadings: 12.5 phr and 25 phr.

The compounds were produced on a laboratory mill at a roll temperature of 20 °C in about 10 minutes.

First, the polymer was rolled into a uniform coat. Then Aktisil Q was added - if contained - and completely incorporated. With a spatula, the respective peroxide was applied to the compound and also incorporated.

To ensure identical production, all compounds were removed from the roll with a scraper, rolled, and then placed back on the roll in a reversed position. This process was repeated 10 times.

The vulcanization took place under the conditions shown in Fig. 3.

All values shown in the following refer to test specimens that were post-cured at 200 °C for 4 hours.

2.2 Tests

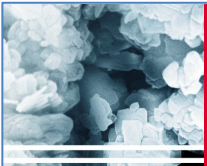
 EINLEITUNG EXPERIMENTELLES ERGEBNISSE ZUSAMMENFASSUNG <u>ANHANG</u> 	Prüfnormen HOFFMANN MINERAL <table> <tr> <th>Prüfung</th><th>Norm</th></tr> <tr> <td>Mooney Viskosität, ML 1+4</td><td>DIN ISO 289-1</td></tr> <tr> <td>Mooney Scorch, ML +5</td><td>DIN ISO 289-2</td></tr> <tr> <td>Rotorloses Vulkameter</td><td>DIN 53 529 Part 3</td></tr> <tr> <td>Härte</td><td>DIN ISO 7619-1</td></tr> <tr> <td>Zugfestigkeit</td><td>DIN 53 504, S2</td></tr> <tr> <td>Spannungswert 100 %</td><td>DIN 53 504, S2</td></tr> <tr> <td>Reißdehnung</td><td>DIN 53 504, S2</td></tr> <tr> <td>Rückprall</td><td>DIN 53 512</td></tr> <tr> <td>Weiterreißwiderstand</td><td>DIN ISO 34-1, A</td></tr> <tr> <td>Druckverformungsrest</td><td>DIN ISO 815-1, B</td></tr> </table> VM-0 01.2020	Prüfung	Norm	Mooney Viskosität, ML 1+4	DIN ISO 289-1	Mooney Scorch, ML +5	DIN ISO 289-2	Rotorloses Vulkameter	DIN 53 529 Part 3	Härte	DIN ISO 7619-1	Zugfestigkeit	DIN 53 504, S2	Spannungswert 100 %	DIN 53 504, S2	Reißdehnung	DIN 53 504, S2	Rückprall	DIN 53 512	Weiterreißwiderstand	DIN ISO 34-1, A	Druckverformungsrest	DIN ISO 815-1, B
Prüfung	Norm																						
Mooney Viskosität, ML 1+4	DIN ISO 289-1																						
Mooney Scorch, ML +5	DIN ISO 289-2																						
Rotorloses Vulkameter	DIN 53 529 Part 3																						
Härte	DIN ISO 7619-1																						
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Rückprall	DIN 53 512																						
Weiterreißwiderstand	DIN ISO 34-1, A																						
Druckverformungsrest	DIN ISO 815-1, B																						

Fig. 5

The values shown in the following diagrams and at the end in the results tables refer to tests performed in accordance with the standards listed in Fig. 5.

3 Results

3.1 Elastosil R 401

3.1.1 Formulation and resulting hardness

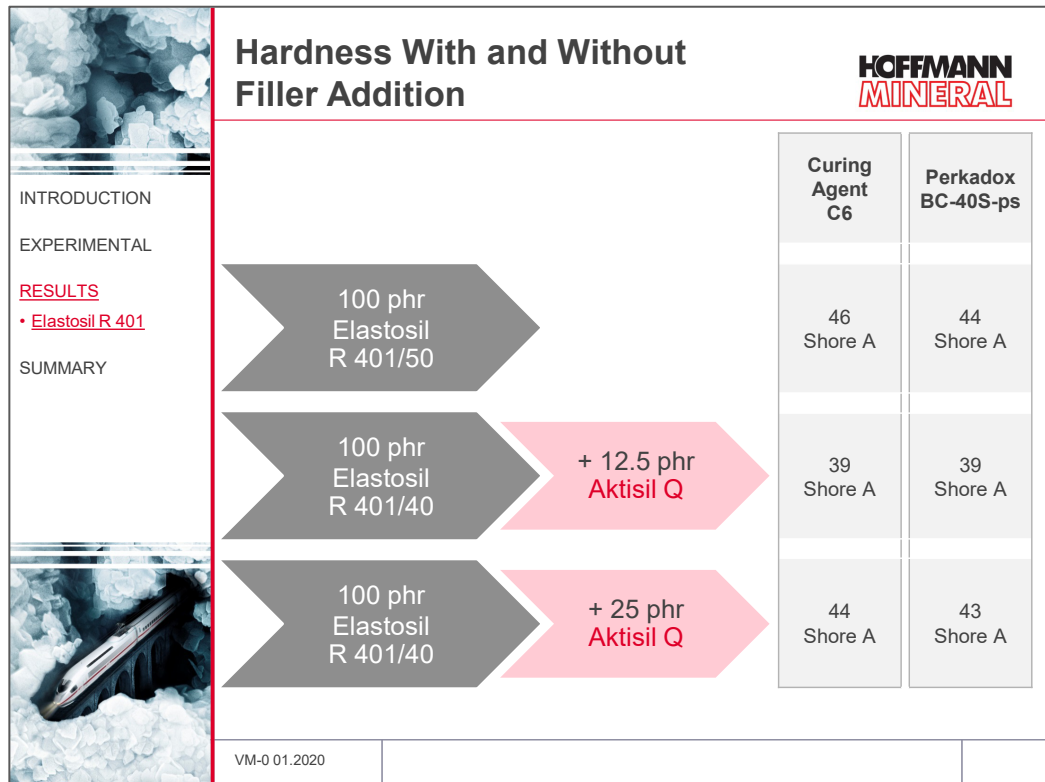


Fig.6

Fig. 6 shows the hardness settings with the two peroxides resulting from the addition of 12.5 phr or 25 phr Aktisil Q to Elastosil R 401/40 in comparison with the values obtained with Elastosil R 401/50.

3.1.2 Mechanical properties – Curing Agent C6

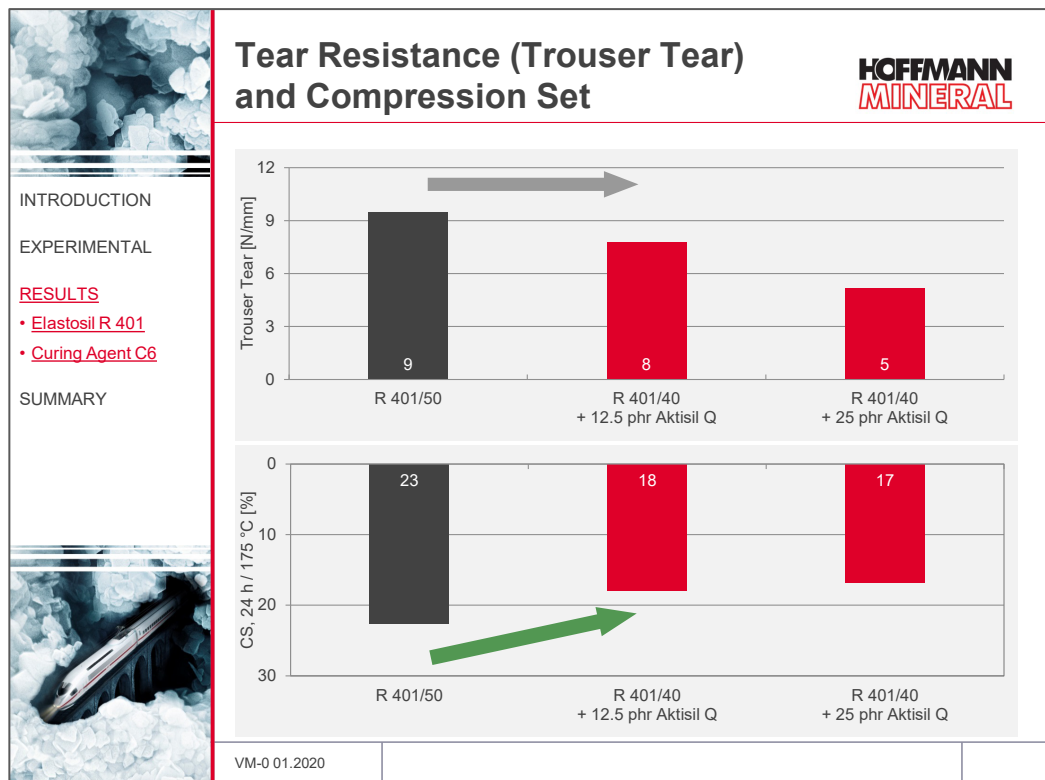


Fig. 7

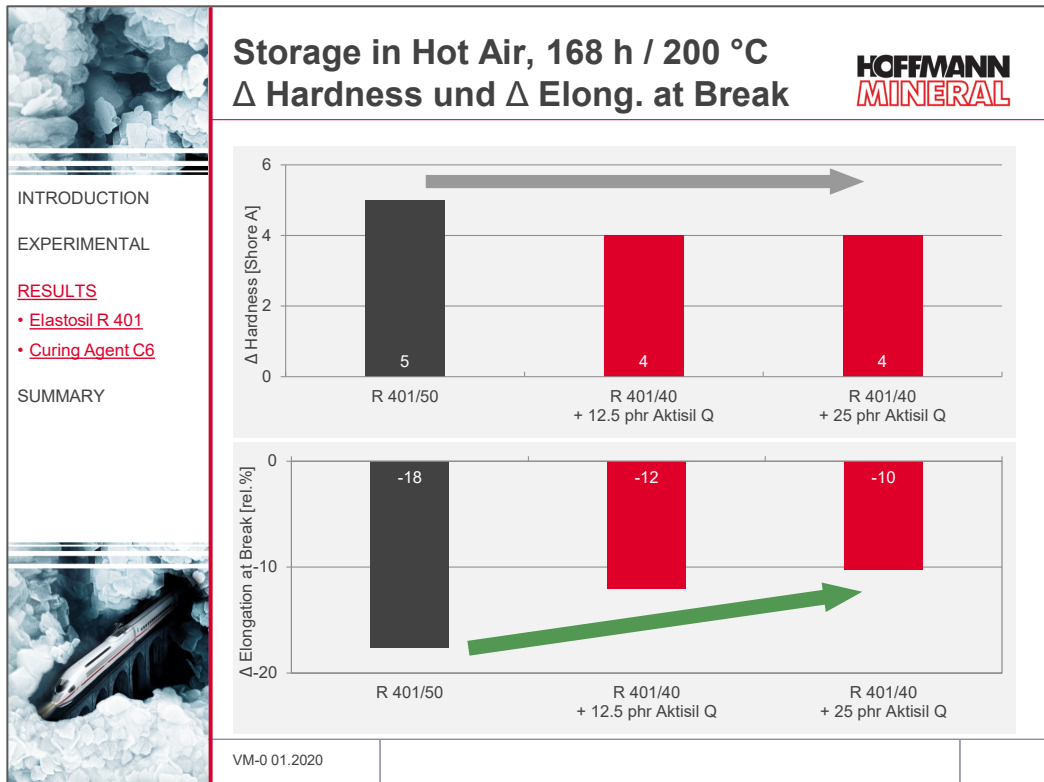


Fig. 8

The addition of 12.5 phr Aktisil Q to Elastosil R 401/40 gives a tear resistance comparable to that of Elastosil R 401/50 without additional filler. At the same time, the compression set decreases (Fig. 7).

In addition, the hot air resistance can be somewhat improved by the use of Aktisil Q, as confirmed by the slightly less pronounced change of elongation at break after storage in hot air (Fig. 8).

3.1.3 Mechanical properties – Perkadox BC-40S-ps

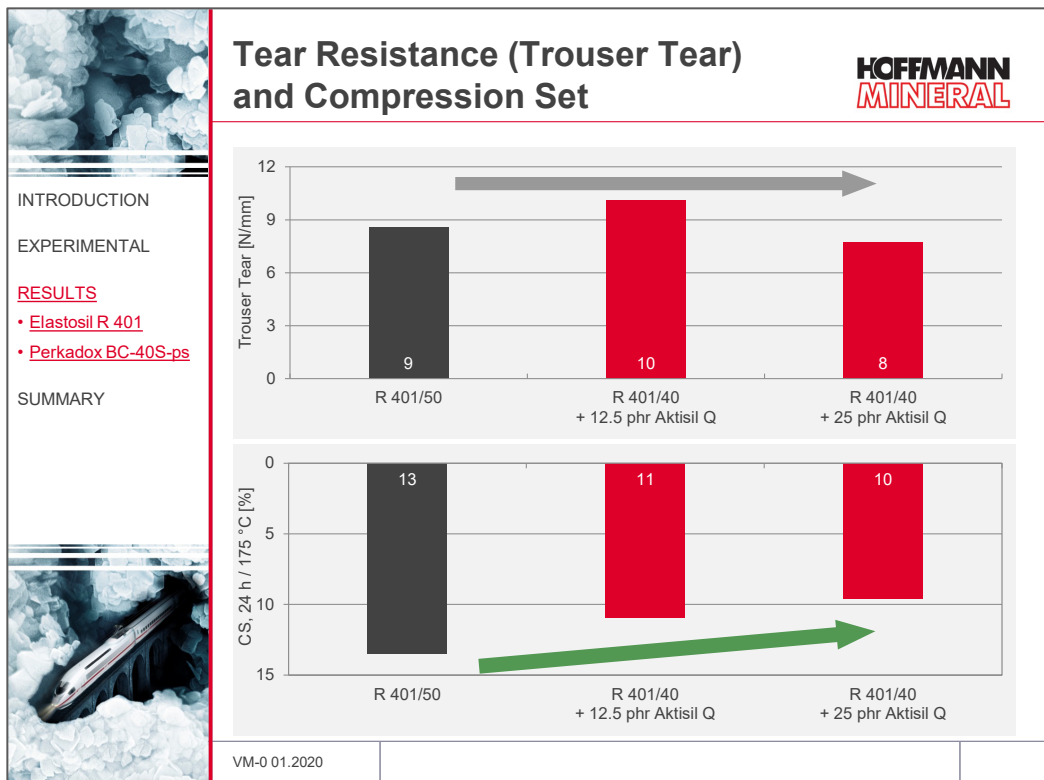


Fig. 9

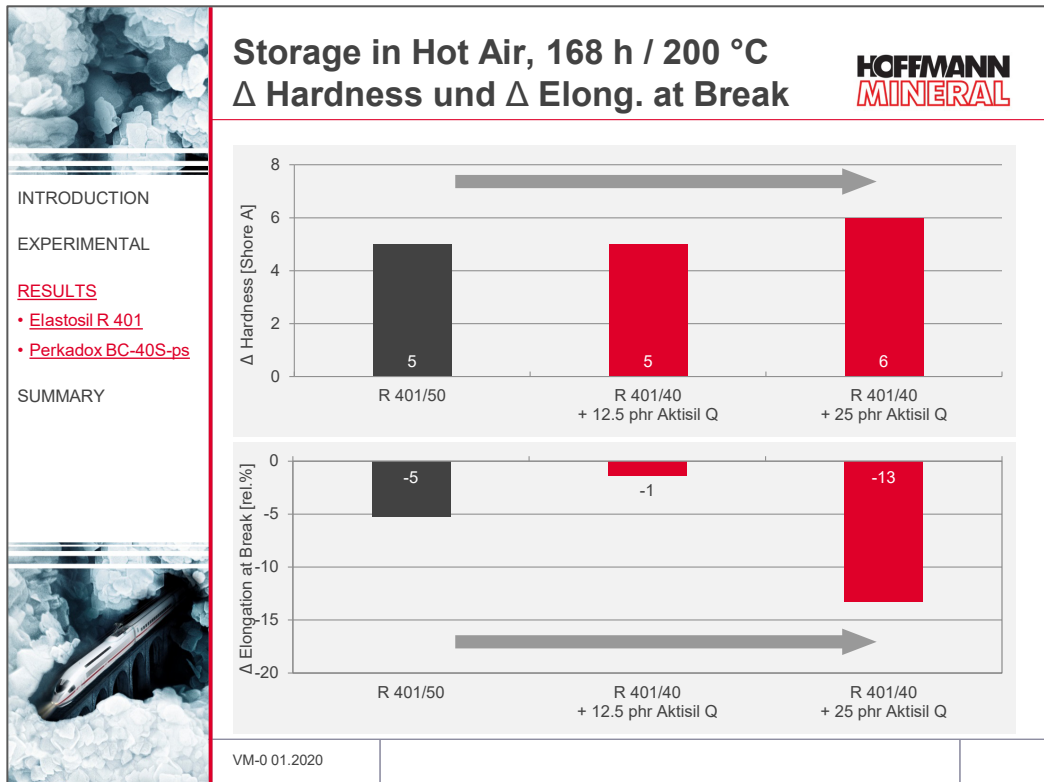


Fig. 10

With Perkadox BC-40S-ps, the tear resistance to Elastosil R 401/50 practically does not change when Aktisil Q is added to Elastosil R 401/40, while the compression set is rather reduced (Fig. 9).

The hot air resistance of Aktisil Q in Elastosil R 401/40 remains comparable to that of Elastosil R 401/50 (Fig. 10).

3.1.4 Compound costs

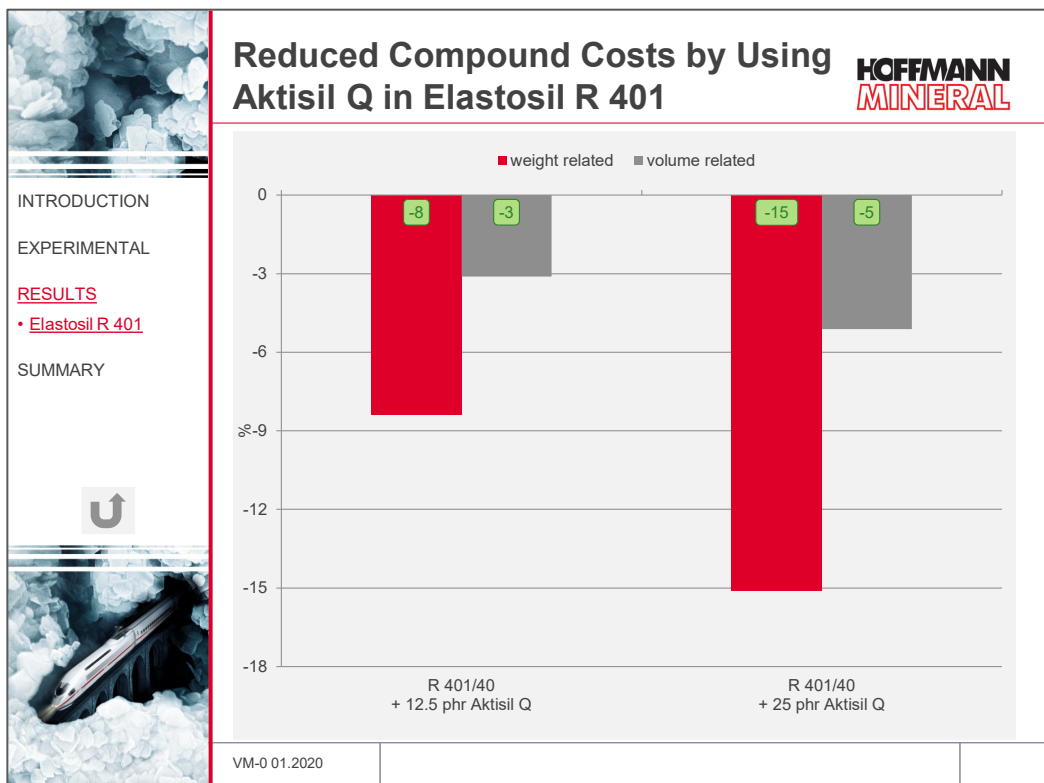


Fig. 11

The more Aktisil Q is added to the polymer, the more the compound costs are reduced (Fig. 11). Thus, 25 phr Aktisil Q can mean a saving of 15 % (based on compound weight), or 5 % (based on compound volume).

3.2 Elastosil R 420

3.2.1 Formulation and resulting hardness

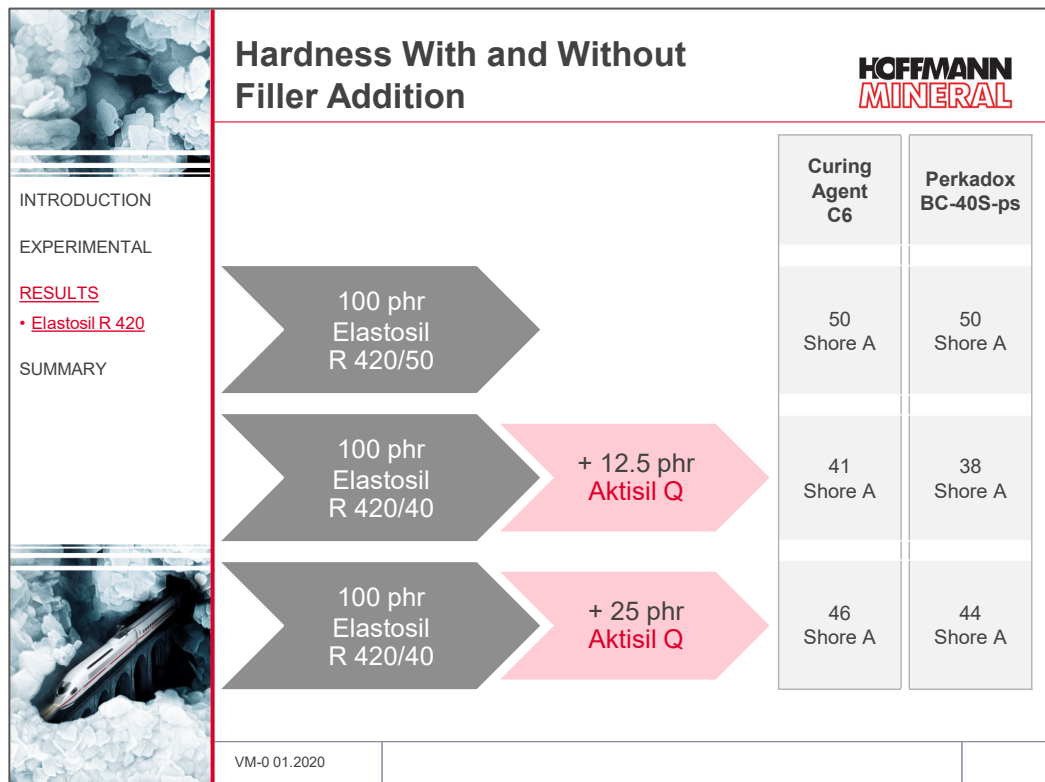


Fig. 12

Fig. 12 shows the hardness settings with the two peroxides resulting from the addition of 12.5 phr resp. 25 phr Aktisil Q to Elastosil R 420/40 in comparison with the values obtained with Elastosil R 420/50.

3.2.2 Mechanical properties – Curing Agent C6

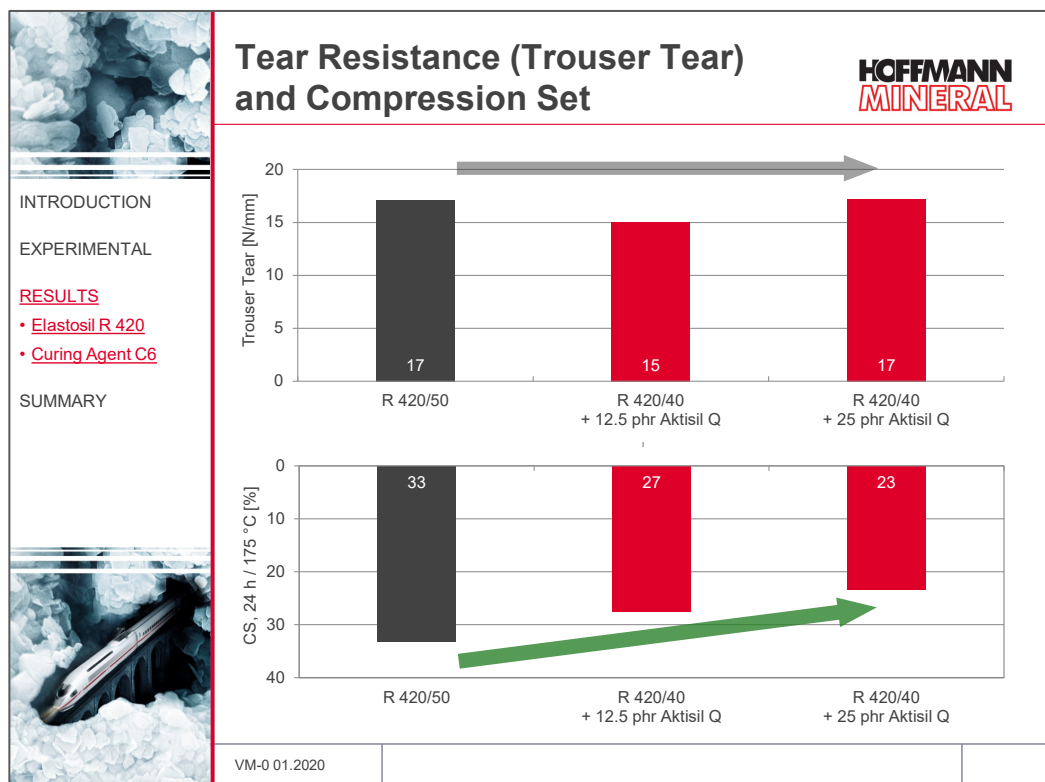


Fig. 13

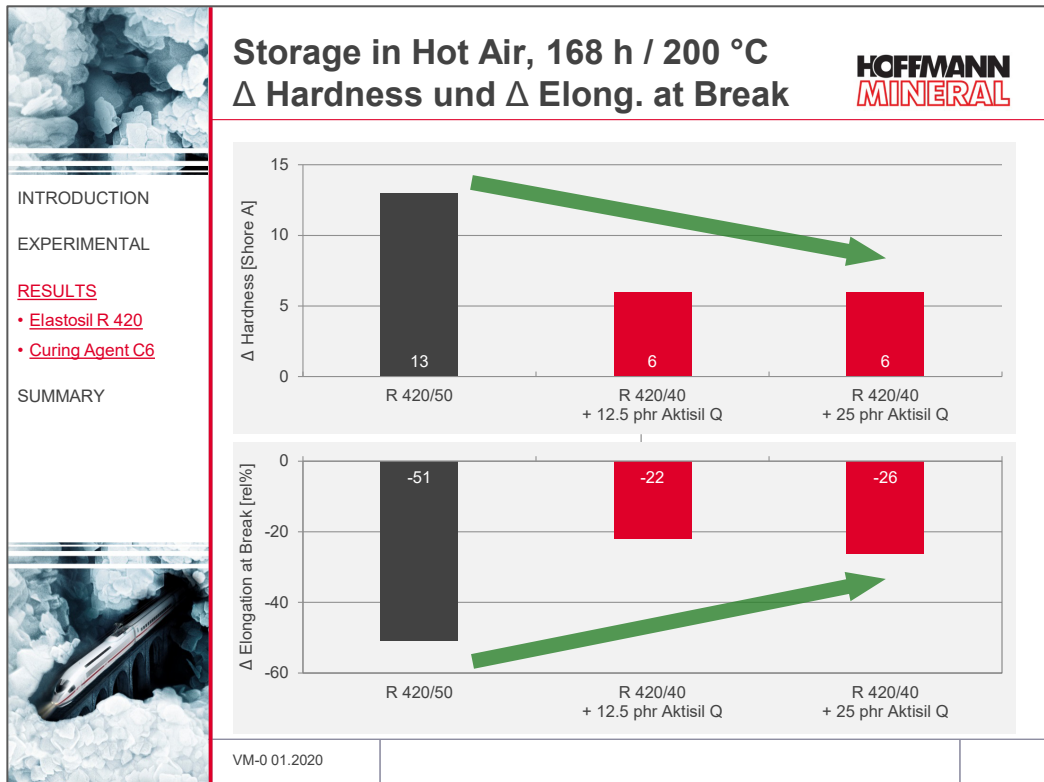


Fig. 14

Fig. 13 shows that when using Curing Agent C6, Aktisil Q can maintain the high tear resistance of Elastosil R 420/50 when added to Elastosil R 420/40. At the same time it significantly reduces the compression set: 25 phr Aktisil Q, for example, will lead to an absolute reduction of 10 %, corresponding to a 30 % improvement.

In addition, the resistance to hot air is considerably improved (Fig. 14). While with Elastosil R 420/50, without additional filler, the hardness increases strongly and elongation at break is reduced by half, Aktisil Q shows its positive effect. Here the hardness increase and the reduction of elongation at break are markedly lower, i.e. an improvement by about 50 %.

3.2.3 Mechanical properties – Perkadox BC-40S-ps

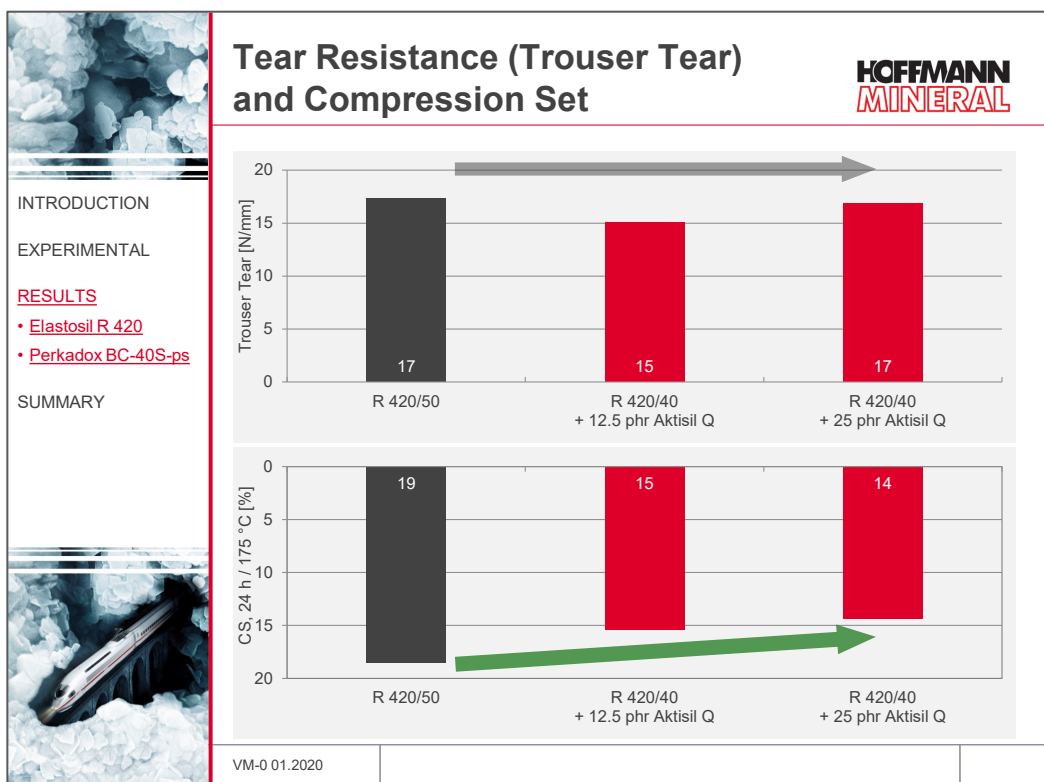


Fig. 15

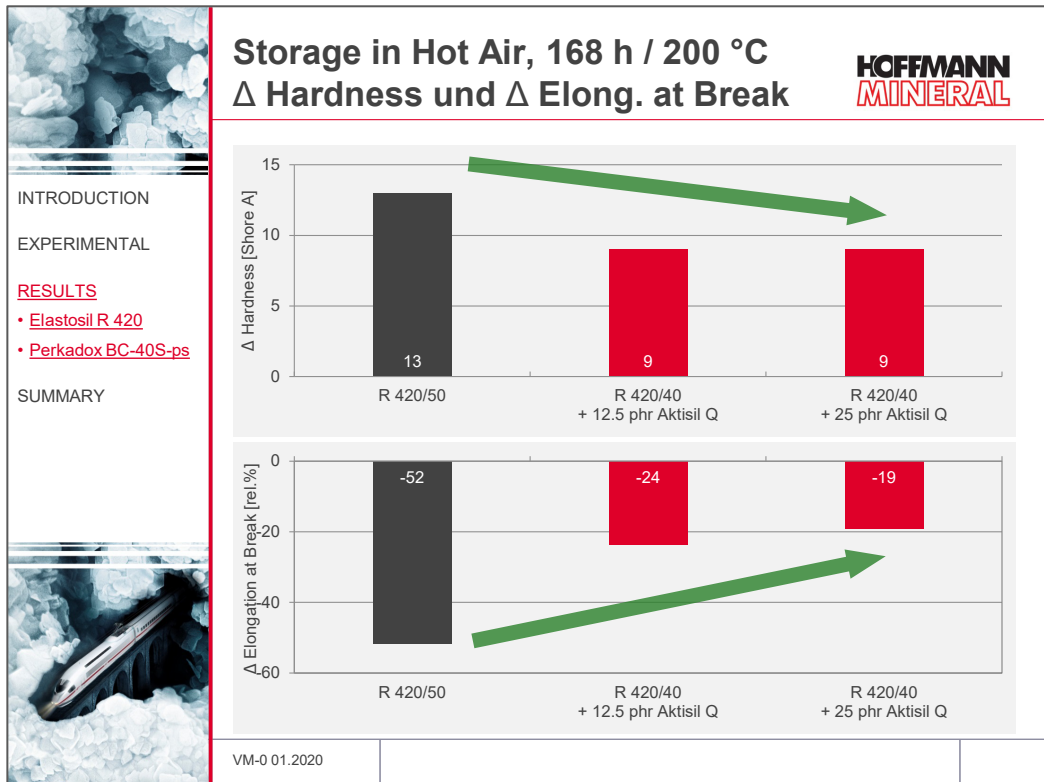


Fig. 16

Elastosil R 420/50 already has a very good compression set when Perkadox BC-40S-ps is used (Fig. 15). Aktisil Q can even improve this good level. 12.5 phr or 25 phr added to Elastosil R 420/40 result in approx. 5 % lower values, corresponding to an improvement of 21 to 26 %. Remarkable is the unchanged high tear resistance.

Also in this polymer the use of Aktisil Q results in a pronounced improved resistance to hot air (Fig. 16). The increase in hardness is lower and elongation at break is also reduced to a much lesser extent. The latter is improved by Aktisil Q up to 63 %.

3.2.4 Compound costs

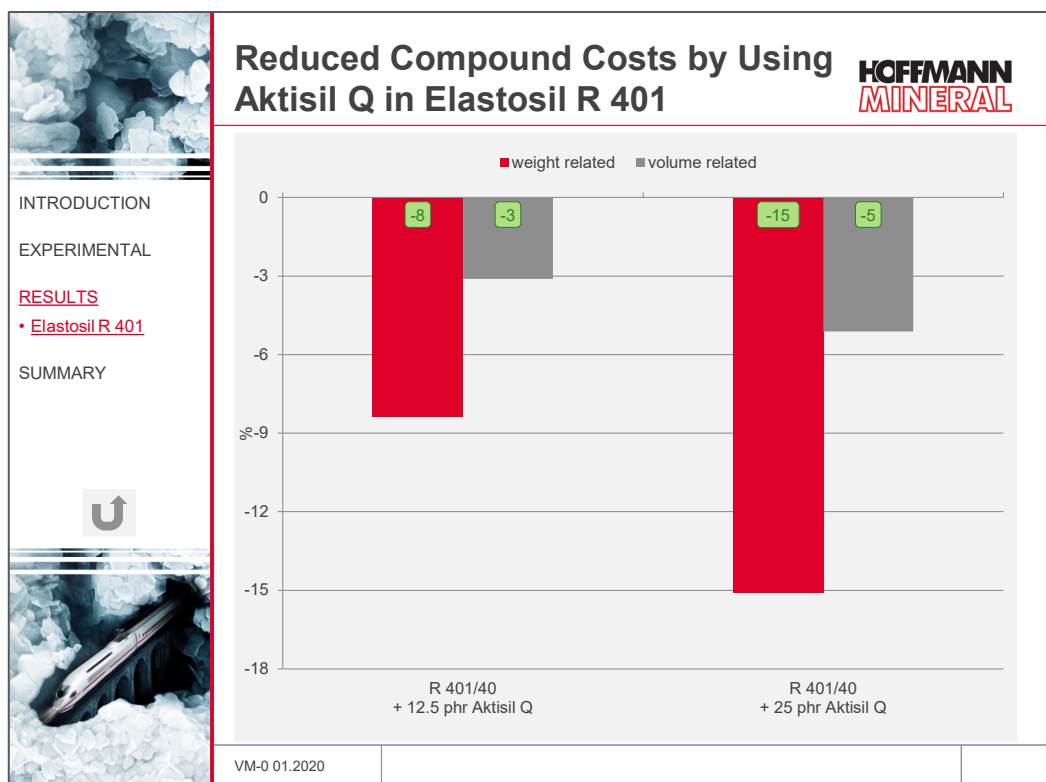


Fig. 17

Already 12.5 phr Aktisil Q in Elastosil R 420/40 can reduce the volume-related compound cost by 6 % compared to Elastosil R 420/50 (Fig. 17). Increasing the dosage to 25 phr results in a further cost reduction of 8 %. In terms of weight savings, Aktisil Q achieves 9 % and 16 % respectively.

3.3 Elastosil R 752

3.3.1 Formulation and resulting hardness

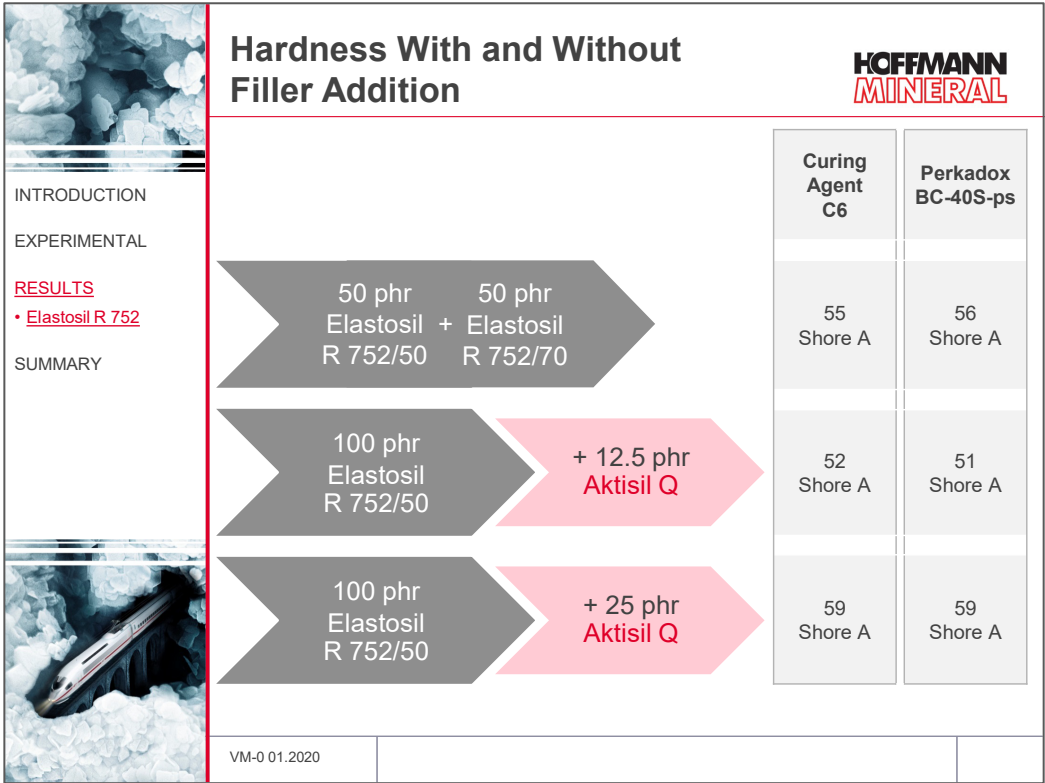


Fig. 18

Fig. 18 shows the hardness settings with the two peroxides resulting from the addition of 12.5 phr or 25 phr Aktisil Q to Elastosil R 752/50. As a comparison without additional filler, the 1:1 combination of Elastosil R 752/50 and Elastosil R 752/70 is used, as a 60 Shore A compound of this series is not offered by the manufacturer.

3.3.2 Mechanical properties – Curing Agent C6

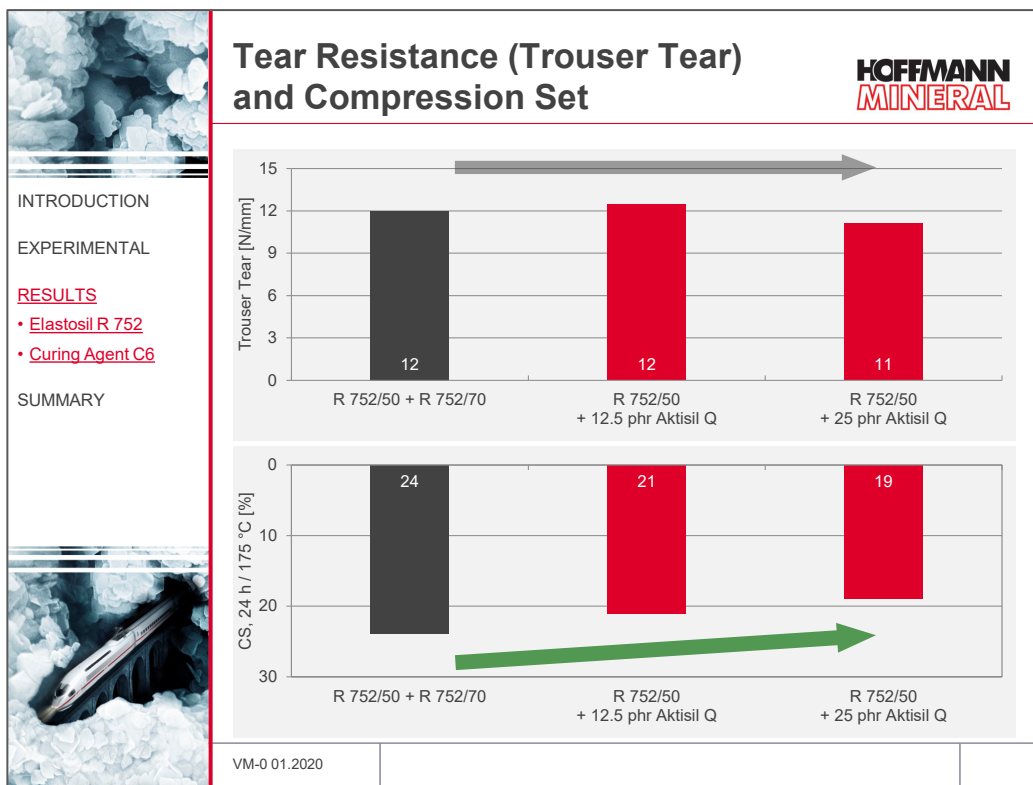


Fig. 19

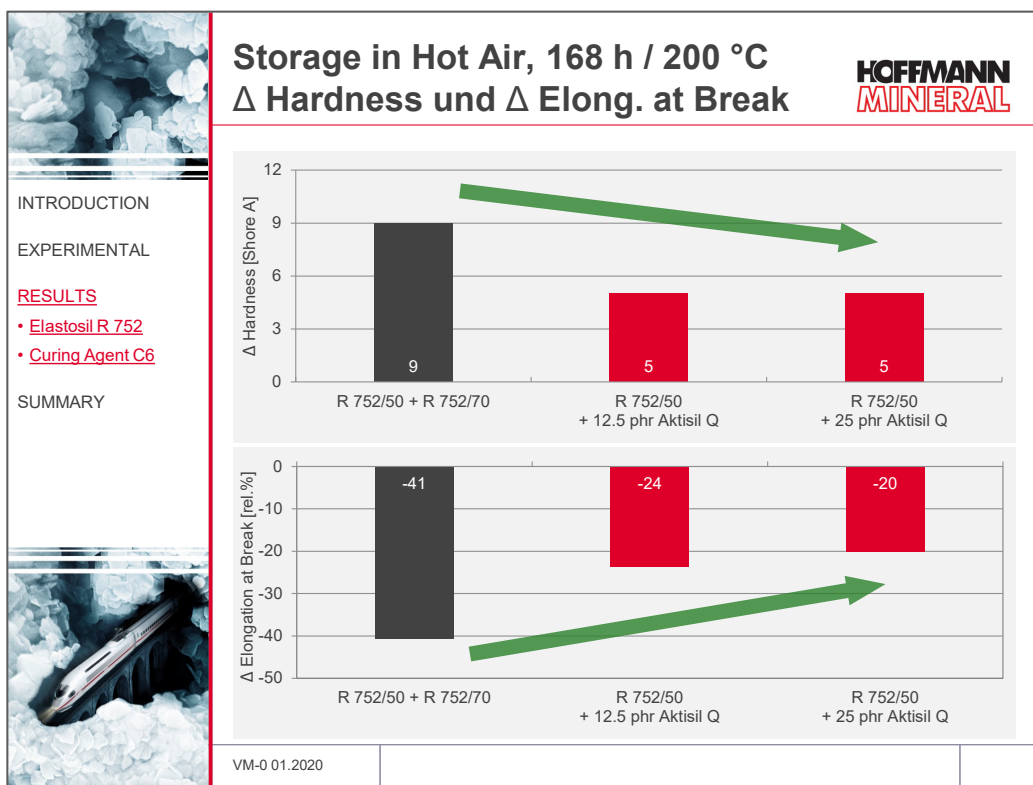


Fig. 20

While with increasing Aktisil Q content the compression set is reduced, the tear resistance remains unchanged (Fig. 19).

After storage in hot air, the hardness with Aktisil Q in Elastosil R 752/50 increases less than with the blend of Elastosil R 752/50 and Elastosil R 752/70 (Fig. 20). Aktisil Q again has a very positive effect on the elongation at break. The improvements achieved are up to about 50 %.

3.3.3 Mechanical properties – Perkadox BC-40S-ps

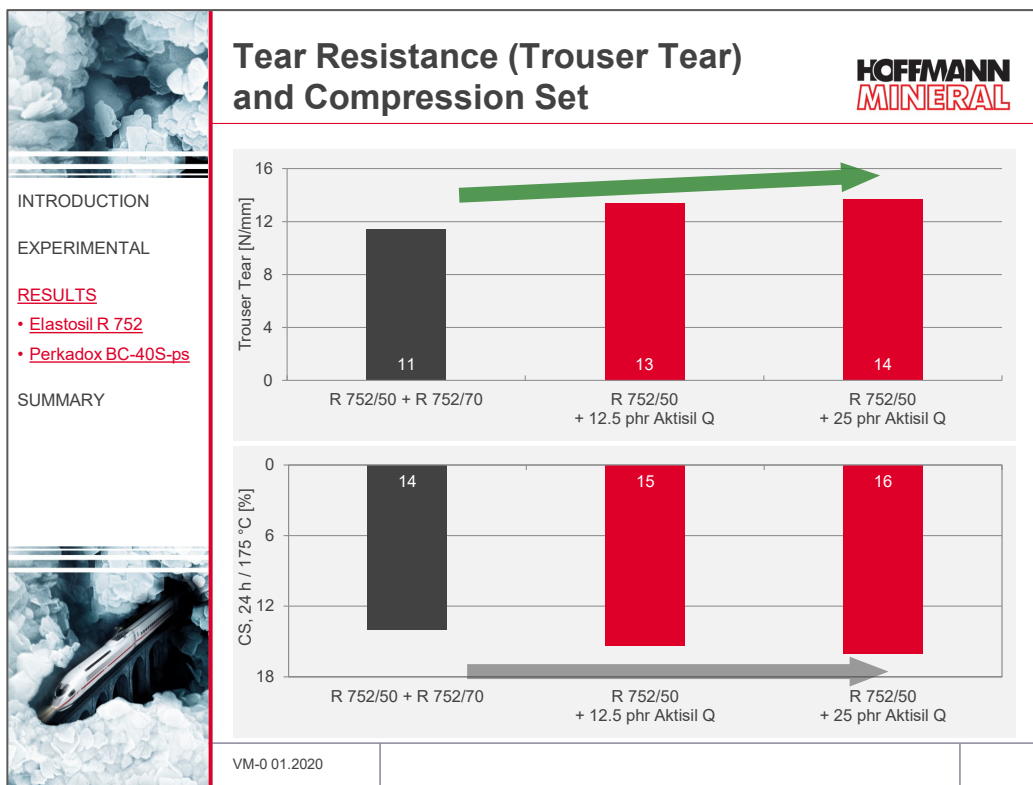


Fig. 21

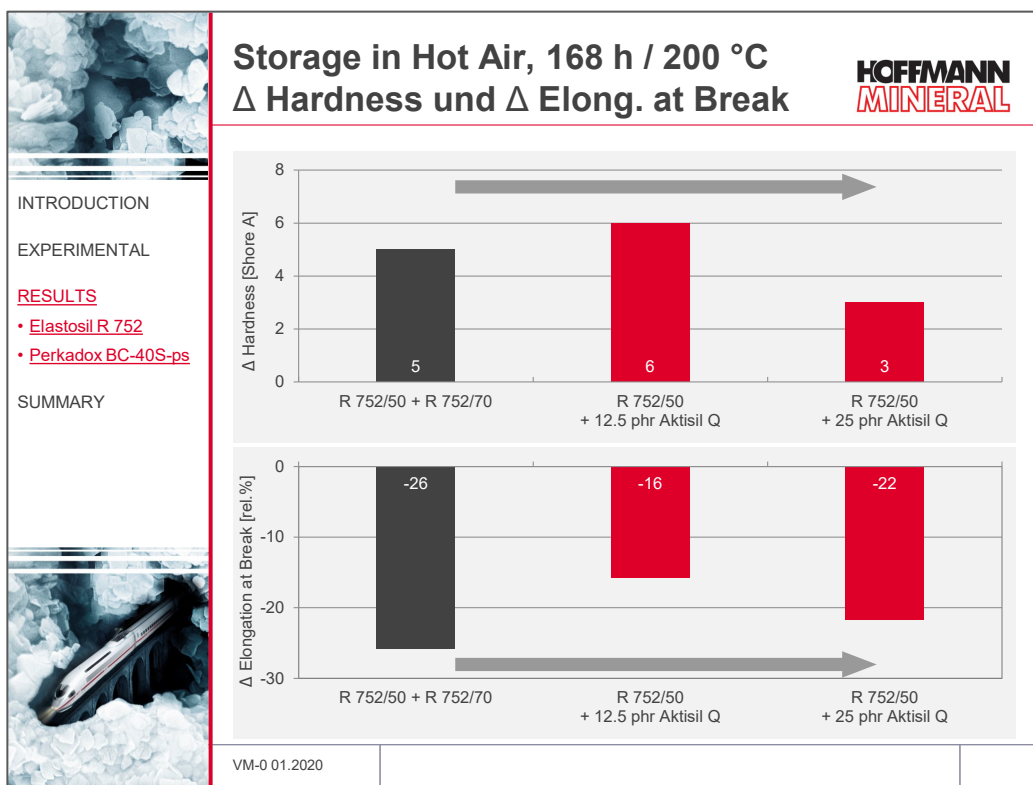


Fig. 22

If Perkadox BC-40S-ps is used in this polymer series, Aktisil Q leads to an increase in tear resistance, while the compression set hardly changes (Fig. 21).

As Fig. 22 shows, the good hot air resistance of the combination of Elastosil R 752/50 and Elastosil R 752/70 is also maintained when Aktisil Q is deployed.

3.3.4 Damping properties

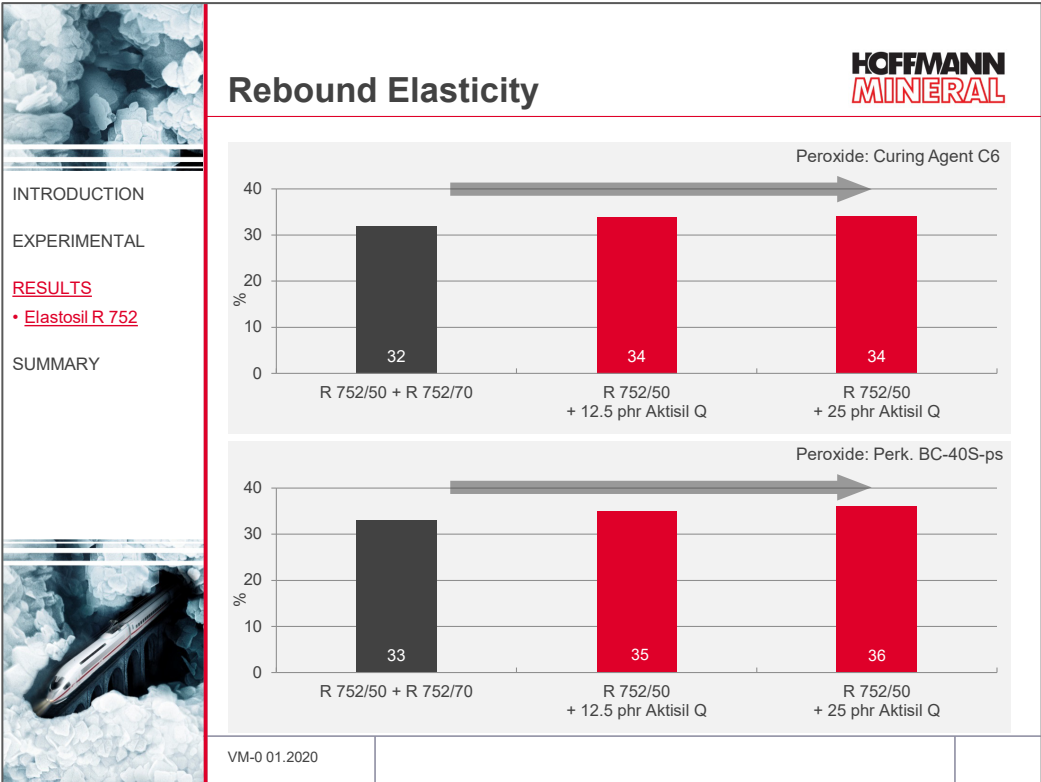


Fig. 23

Fig. 23 shows that the damping properties of the Elastosil R 752 series are maintained even after the addition of Aktisil Q.

3.3.5 Compound costs

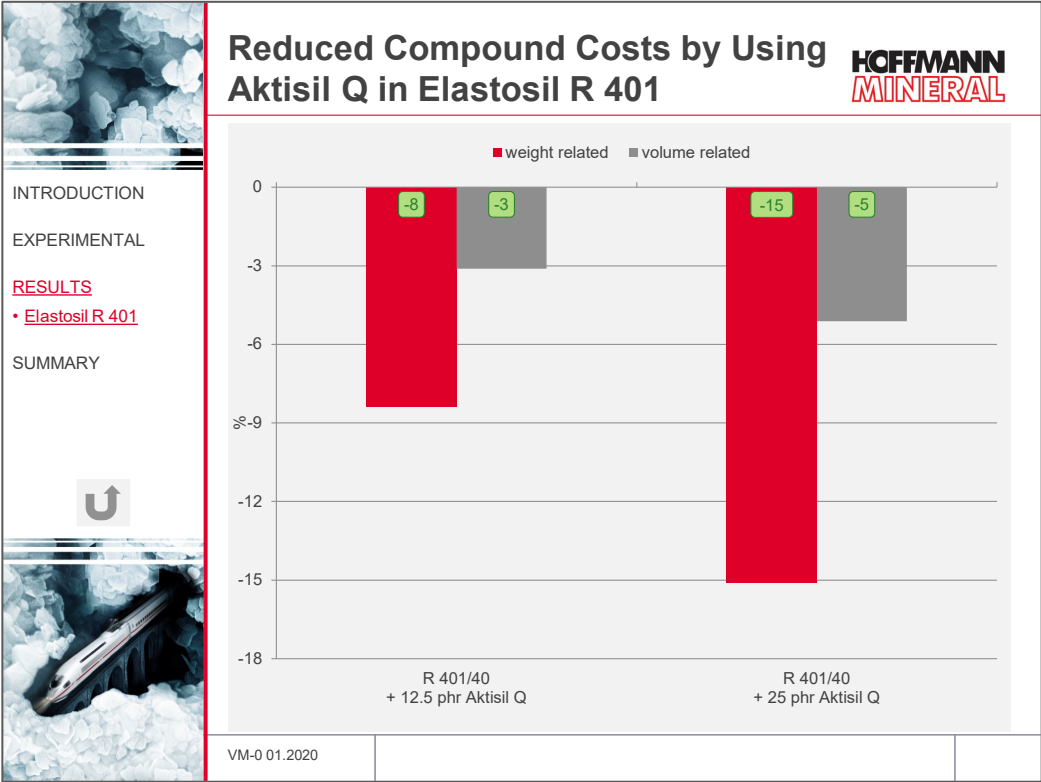


Fig. 24

In Elastosil R 752, the use of Aktisil Q results in a reduction of the volume-related compound cost by 6 % or 10 %, depending on the dosage. The respective weight-related savings are 10 % and 18 %.

The results of this investigation clearly show that Aktisil Q can be used to improve the performance of different silicone polymers, often even to optimize contradictory properties such as compression set and tear resistance at the same time. In addition, processing is greatly facilitated, as Aktisil Q reduces tack. Furthermore, costs can be reduced, in part significantly.

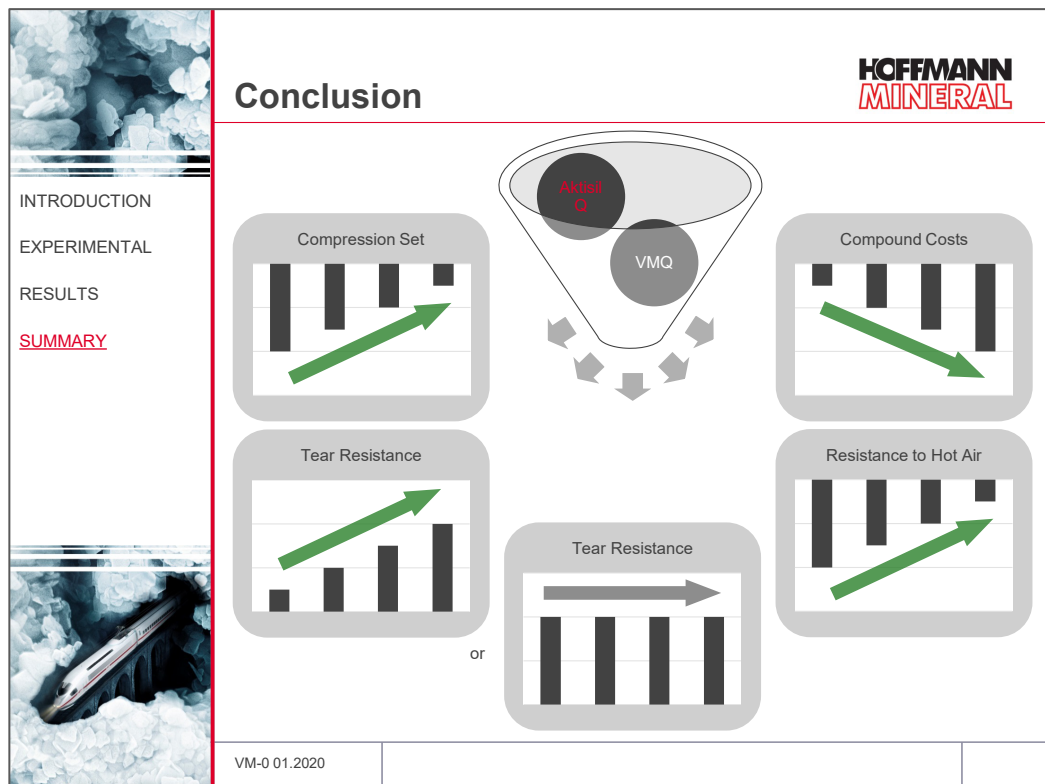


Fig. 25

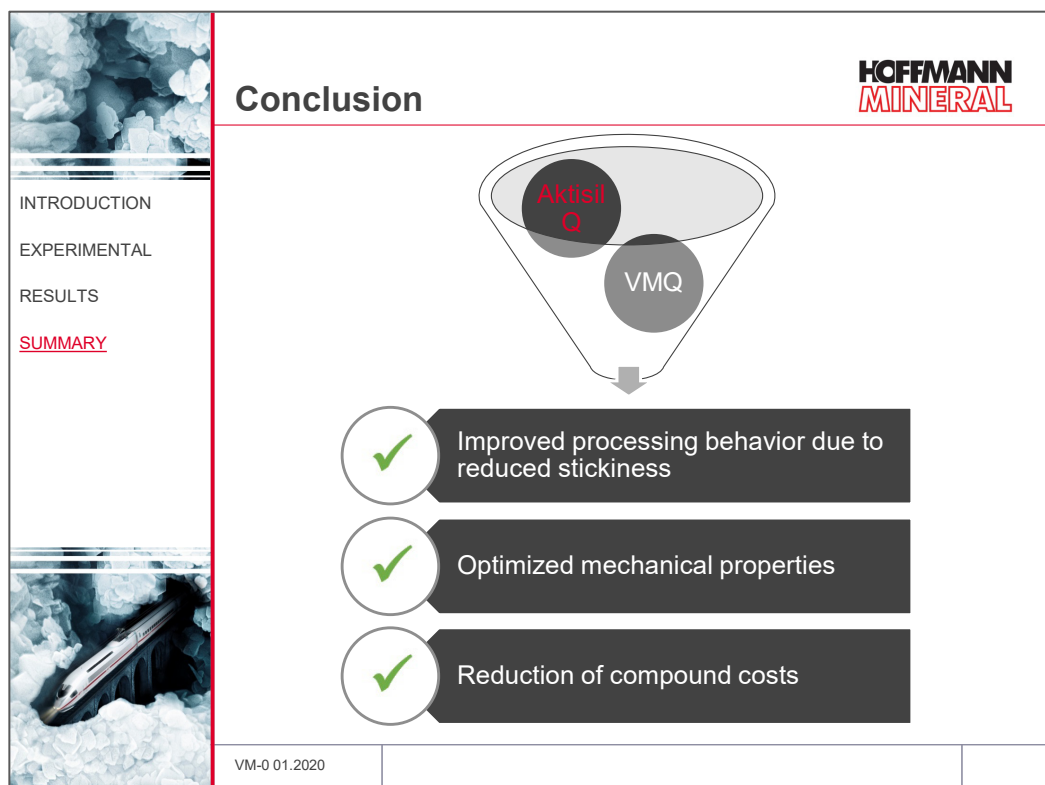
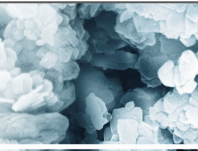
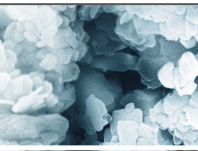
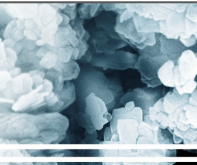


Fig. 26

Although our technical application advice and the information in this report are based on experience and provided to the best of our knowledge, they should only be construed as non-binding and are not in any way guaranteed. Claims cannot be derived from utilizing our data and recommendations, given that we have no influence over the working environment or conditions of use. Moreover, we can accept no liability whatsoever for any patent infringements that might result from utilization of the information we have provided.

 INTRODUCTION EXPERIMENTAL RESULTS SUMMARY APPENDIX			Curing Agent C6			Perkadox BC-40S-ps		
			R 401/50	R 401/40 + 12.5 phr Aktisil Q	R 401/40 + 25 phr Aktisil Q	R 401/50	R 401/40 + 12.5 phr Aktisil Q	R 401/40 + 25 phr Aktisil Q
	Rheology							
	Mooney Viscosity, ML 1+4, 120 °C	MU	14	11	13	14	11	13
	Mooney Scorch, ML +5, 120 °C	min.	72	20	12	64	17	10
	Temperature Curemeter	°C	165			180		
	Rotorless Curemeter M _{min}	Nm	0.04	0.03	0.03	0.04	0.03	0.03
	Rotorless Curemeter V _{max}	Nm/min.	0.26	0.23	0.26	0.76	0.64	0.76
	Rotorless Curemeter t ₉₀	min.	2.7	2.5	2.3	1.0	0.9	0.9
	Mechanical properties – post-cured, 4 h / 200 °C							
	Hardness	Sh. A	46	39	44	44	39	43
	Tensile strength	MPa	13	10	10	12	9.2	9.6
	Modulus 100 %	MPa	0.9	0.7	1.0	0.9	0.8	1.0
	Elongation at Break	%	679	702	631	647	624	616
	Tear resistance	N/mm	9.5	7.8	5.2	8.6	10	7.8
	Compression Set, 24 h / 175 °C, 25 % Def.	%	23	18	17	13	11	9.6
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 INTRODUCTION EXPERIMENTAL RESULTS SUMMARY APPENDIX			Curing Agent C6			Perkadox BC-40S-ps		
			R 401/50	R 401/40 + 12.5 phr Aktisil Q	R 401/40 + 25 phr Aktisil Q	R 401/50	R 401/40 + 12.5 phr Aktisil Q	R 401/40 + 25 phr Aktisil Q
	Mechanical properties after storage in hot air (post-cured specimens), 168 h / 200 °C							
	Hardness	Sh. A	51	43	48	49	44	49
	Tensile strength	MPa	11	8.9	8.5	11	9.5	8.5
	Modulus 100 %	MPa	1.3	09	1.3	1.1	1.0	1.2
	Elongation at Break	%	560	617	567	614	616	535
	Δ Hardness	Sh. A	+5	+4	+4	+5	+5	+6
	Δ Tensile strength	%	-17	-15	-16	-9	+4	-12
	Δ Modulus 100 %	%	+40	+25	+26	+16	+23	+19
	Δ Elongation at break	%	-18	-12	-10	-5	-1	-13
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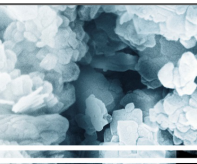


Table of Results

Elastosil R 420



		Curing Agent C6			Perkadox BC-40S-ps		
		R 420/50	R 420/40 + 12.5 phr Aktisil Q	R 420/40 + 25 phr Aktisil Q	R 420/50	R 420/40 + 12.5 phr Aktisil Q	R 420/40 + 25 phr Aktisil Q
Rheology							
Mooney Viscosity, ML 1+4, 120 °C	MU	17	14	15	17	14	16
Mooney Scorch, ML +5, 120 °C	min.	57	20	11	55	16	8,4
Temperature Curemeter	°C	165			180		
Rotorless Curemeter M _{min}	Nm	0.04	0.03	0.04	0.04	0.03	0.03
Rotorless Curemeter V _{max}	Nm/min.	0.11	0.12	0.18	0.41	0.38	0.51
Rotorless Curemeter t ₉₀	min.	4.4	4.1	3.6	1.5	1.3	1.1
Mechanical properties – post-cured, 4 h / 200 °C							
Hardness	Sh. A	50	41	46	50	38	44
Tensile strength	MPa	10	8.8	8.2	9.5	8.9	7.9
Modulus 100 %	MPa	1.5	1.0	1.3	1.5	1.0	1.3
Elongation at Break	%	651	705	630	642	725	614
Tear resistance	N/mm	17	15	17	17	15	17
Compression Set, 24 h / 175 °C, 25 % Def.	%	33	27	23	19	15	14
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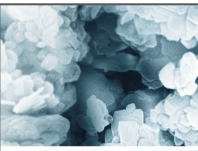
Table of Results

Elastosil R 420



		Curing Agent C6			Perkadox BC-40S-ps		
		R 420/50	R 420/40 + 12.5 phr Aktisil Q	R 420/40 + 25 phr Aktisil Q	R 420/50	R 420/40 + 12.5 phr Aktisil Q	R 420/40 + 25 phr Aktisil Q
Mechanical properties after storage in hot air (post-cured specimens), 168 h / 200 °C							
Hardness	Sh. A	63	47	52	63	47	53
Tensile strength	MPa	9,5	7,8	6,8	8,5	7,5	7,0
Modulus 100 %	MPa	3,2	1,4	1,8	3,1	1,3	1,7
Elongation at Break	%	320	551	464	310	553	496
Δ Hardness	Sh. A	+13	+6	+6	+13	+9	+9
Δ Tensile strength	%	-7	-11	-17	-10	-16	-11
Δ Modulus 100 %	%	+116	+38	+35	+115	+34	+29
Δ Elongation at break	%	-51	-22	-26	-52	-24	-19

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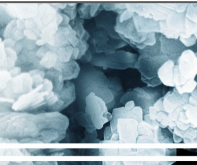
Table of Results

Elastosil R 752

HOFFMANN

MINERAL

		Curing Agent C6			Perkadox BC-40S-ps		
		R 752/50 + R 752/70	R 752/50 + 12.5 phr Aktisil Q	R 752/50 + 25 phr Aktisil Q	R 752/50 + R 752/70	R 752/50 + 12.5 phr Aktisil Q	R 752/50 + 25 phr Aktisil Q
Rheology							
Mooney Viscosity, ML 1+4, 120 °C	MU	24	24	27	25	25	26
Mooney Scorch, ML +5, 120 °C	min.	64	23	13	66	22	13
Temperature Curemeter	°C	165			180		
Rotorless Curemeter M _{min}	Nm	0.09	0.10	0.11	0.08	0.09	0.10
Rotorless Curemeter V _{max}	Nm/min.	0.32	0.33	0,50	0.79	0.77	0.96
Rotorless Curemeter t ₉₀	min.	4.9	4.5	3.2	1.5	1.5	1.3
Mechanical properties – post-cured, 4 h / 200 °C							
Hardness	Sh. A	55	52	59	56	51	59
Tensile strength	MPa	9.6	8.8	7.3	9.5	8.6	7.8
Modulus 100 %	MPa	1.2	1.1	1.4	1.2	1.2	1.5
Elongation at Break	%	672	698	557	637	631	543
Tear resistance	N/mm	12	12	11	11	13	14
Rebound Elasticity	%	32	34	34	33	35	36
Compression Set, 24 h / 175 °C, 25 % Def	%	24	21	19	14	15	16
VM-0 01.2020							



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Table of Results

Elastosil R 752

HOFFMANN

MINERAL

		Curing Agent C6			Perkadox BC-40S-ps		
		R 752/50 + R 752/70	R 752/50 + 12.5 phr Aktisil Q	R 752/50 + 25 phr Aktisil Q	R 752/50 + R 752/70	R 752/50 + 12.5 phr Aktisil Q	R 752/50 + 25 phr Aktisil Q
Mechanical properties after storage in hot air (post-cured specimens), 168 h / 200 °C							
Hardness	Sh. A	64	57	64	61	57	62
Tensile strength	MPa	5.5	6.1	5.2	6.2	7.0	6.1
Modulus 100 %	MPa	1.6	1.3	1.6	1.5	1.3	1.8
Elongation at Break	%	398	532	445	472	532	425
Δ Hardness	Sh. A	+9	+5	+5	+5	+6	+3
Δ Tensile strength	%	-42	-30	-28	-35	-18	-22
Δ Modulus 100 %	%	+42	+17	+16	+27	+16	+16
Δ Elongation at break	%	-41	-24	-20	-26	-16	-22

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